Influence of temperature on the properties of fireproof materials

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Abstract. The results of experimental studies of the behavior of fireproof materials of Russian production – OGNEZA-LTU, OGNEZA-M-KOR, OG-NEZA-M-KOR (NG), «OGNEBAZALT» PMBOR, OGNEZA-GT under thermal action are presented. Research method – synchronous thermal analysis TG/DCS/dDSC, including differential scanning calorimetry (DSC) and thermogravimetry (TG). Experimental conditions on the NETZSCH thermal analyzer: measurement mode – TG/DCS/dDSC; heating rate: 20 °C /min; heating – up to 1000 °C; atmosphere – N_2. The rates of weight loss of the investigated fire-retardant materials have been studied. A high rate of weight loss was established for the OGNEZA-LTU samples (20.5 %/ min at 335 °C) and OGNEZA-GT (11.9 %/min at 369 °C); low – OGNEZA-M-KOR (NG) – 1.7 %/min at 349 °C, OGNEZA-M-KOR – 6.1%/min at 341 °C, «OGNEBASALT» PMBOR - 3.5 %/min at 388 °C. Time intervals of weight loss were determined: all test samples had maximum weight loss in the range of 15-20 minutes. The temperature ranges of the maximum rate of weight loss were determined: 349 - 388 °C. The best heat-resistant properties were shown by OGNEZA-M-KOR (NG), «OGNEBASALT» PMBOR, OGNEZA-M-KOR. It is proposed to consider the revealed properties when using fire retardant materials for the constructive fire hazard of civil and industrial facilities.

1 Introduction

An urgent task of our time is the production and use of fireproof materials to increase the stability of buildings and structures in the event of a fire and the accumulation of knowledge about the behavior of materials under temperature exposure. The thermal properties of fire retardant materials are important parameters for use on a specific protected object.

Research work on fire-resistant materials is mainly aimed at studying the properties of fire retardants, the mechanism of retarding combustion, pyrolysis and combustion products of materials, increasing the fire resistance of materials, assessing the flammability and fire-retardant efficiency of coatings for metal structures, intumescent fire-retardant materials [1-12]. However, data on thermal properties, especially on the rate of weight loss of Russian-made fire-resistant materials, are scarce [13-18].

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Purpose of the research: to study the effect of temperature on the properties of Russian-made fire-retardant materials.

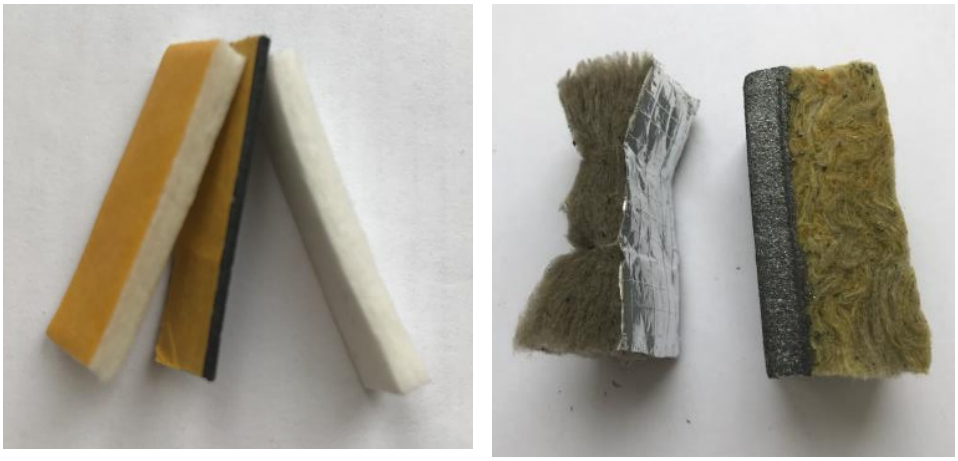
Research objectives:

- study of the rate of weight loss of fire retardant materials;
- determination of temperature ranges for weight loss;
- determination of materials with the best heat-resistant properties.

Characteristics of the research object.

The object of the study was samples of Russian-made fire-retardant materials – Ogneza LLC, St. Petersburg, and OGNEBAZALT MSK LLC, Balashikha, Moscow region (Figure 1):

- fire-fighting tape OGNEZA-LTU and seals: OGNEZA-M-KOR and OG-NEZA-M-KOR (NG);
- fire retardant material «OGNEBASALT»PMBOR;
- fire retardant thermo-expanding sealant OGNEZA-GT in the form of mineral wool heat-insulating plate.



a) *b)*

a is from left to right – self-adhesive sealant OGNEZA-M-KOR 3×10 (thickness, mm×width, mm); heat sealing tape OGNEZA-LTU 1.5×10; seal OGNEZA-M-KOR (NG) 3×10); b is from left to right – «OGNEBASALT» PMBOR (thickness – 8 mm); sealant OGNEZA-GT (thickness – 30 mm).

Fig. 1. Investigated samples of fire retardant materials

Thermo-sealing tape OGNEZA-LTU is designed to fill the voids, gaps between the fire door frame and the door leaf to block the penetration of smoke and fire into adjacent rooms. Fireproof seals-whether OGNEZA-M-KOR, OGNEZA-M-KOR (NG) are used to fire air ducts and equipment insulation. Fire-retardant basalt material PMBOR is used for fire protection of building structures, air ducts, thermal insulation of pipelines, equipment, residential and industrial buildings and structures, attic floors of houses. Fire retardant thermally expanding sealant OGNEZA-GT in the form of a mineral wool heat-insulating plate has found application for fire protection and thermal insulation of reinforced concrete floor slabs, for sealing construction joints, cable and ventilation passages of communications through walls and ceilings.

According to the manufacturers, the OGNEZA-LTU thermal sealing tape is made of rubber, thermally expanding graphite, flame retardant mixture, PVC adhesive tape, seals OGNEZA-M-KOR, OGNEZA-M-KOR (NG) – from silica; «OGNEBASALT» PMBOR and OGNEZA-GT – from basalt rocks.

2 Methods

To study the effect of temperature on the properties of fire retardant materials, the method of synchronous thermal analysis (STA) (GOST R 55134-2012) was used, including differential scanning calorimetry (DSC) and thermogravimetry (TG). The study by the STA method was carried out at the Department of Fire Safety of the Federal State Budgetary Educational Institution of Higher Education «Academy of Civil Protection» on a thermal analyzer STA 449 F3 Jupiter of the German company «NETZSCH».

Characteristics of the experimental conditions: measurement mode – TG/ DCS/dDSC; heating rate: 20 °C/min; heating – up to 1000 °C; atmosphere – N_2.

3 Results and Discussion

Figures 2-6 show the dTG curves of the studied fire-retardant materials, reflecting the rate of decrease in the mass of the samples over time.

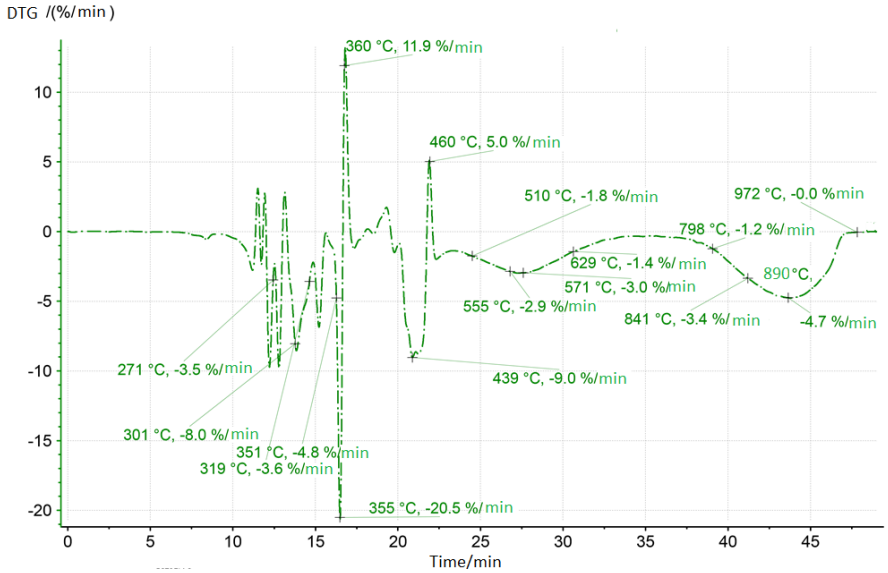


Fig. 2. The rate of change in the mass of the sample of the tape of thermosealing OGNEZA-LTU under thermal action (DTG, %/min)

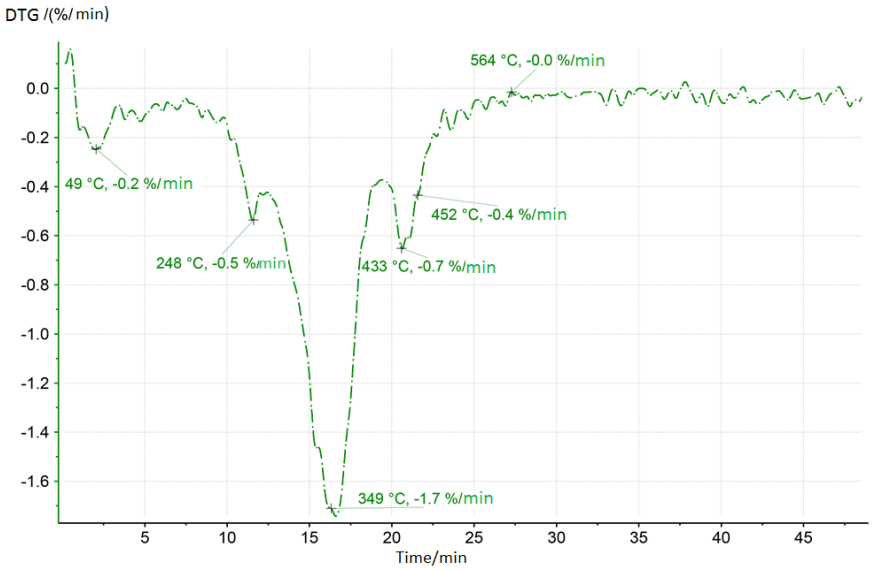


Fig. 3. The rate of change in the mass of the sample sealant OGNEZA-M-KOR (NG) with thermal exposure (DTG,% / min).

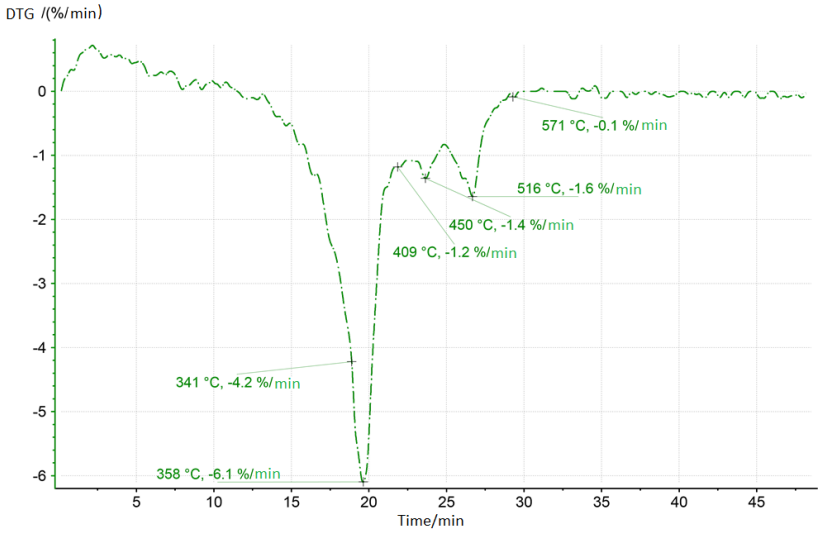


Fig. 4. The rate of change in the mass of the sample sealant OGNEZA-M-KOR with thermal exposure (DTG, % / min)

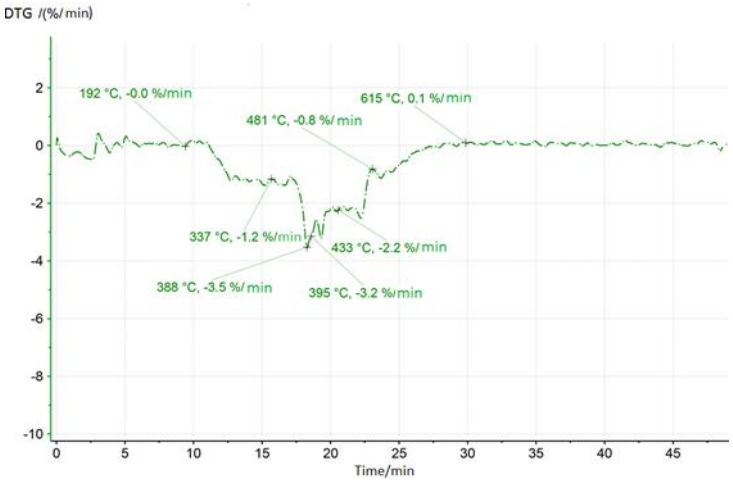


Fig. 5. The rate of change in the mass of the sample of the fire-retardant material «OGNEBASALT» PMBOR under thermal action (DTG, % / min)

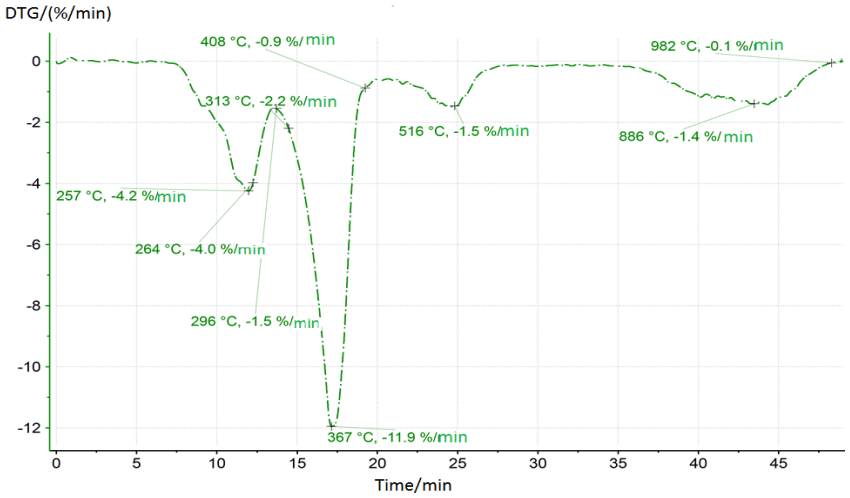


Fig. 6. The rate of change in the mass of the sample of the fire-retardant thermally expanding sealant OGNEZA-GT under thermal action (DTG, % /min)

As can be seen from figures 2-6, the behavior of fire-retardant materials under temperature exposure differs from each other; however, the non-combustible properties of the OGNEZA-M-KOR (NG) seal and the «OGNEBAZALT» PMBOR fire retardant material, declared by the manufacturers as non-combustible, are confirmed material.

Analysis of the curves in Figures 2-6 allows us to draw the following conclusions.

After heating the OGNEZA-LTU sealant sample (Figure 2) from 271 to 351 °C at 11-16 minutes of testing, an increase and decrease in the sample mass are observed in a chaotic manner, which can be explained by the process of swelling and burnout of the organic components of the sealant composition in the resulting loose material structure. The maximum rate of sample weight loss – 20.5%/min occurs when the temperature reaches 355 °C after 16 minutes of fire exposure. At a temperature of 360 °C, the sample is likely to swell with an increase in its mass at a rate of 11.9%/min. At 21-minutes of fire exposure at a temperature of 439 °C, the organic component of the sample burns out at a rate of 9.0 %/min and then swells at a temperature of 460 °C. Subsequent changes in the rate of weight

loss fluctuate in the range of 1.8 - 4.7%/min. Thus, by the 14-th minute of testing and reaching 301 °C, the OGNEZA-LTU sealant will perform protective functions. The swelling of the sample in conjunction with a smooth decrease in mass can be attributed to the advantage of the material (there is no instantaneous destruction of the material, respectively, the decrease in protective functions from hazardous fire factors is gradual).

After heating the OGNEZA-M-KOR (NG) seal sample (Figure 3), the mass loss rate varies from a minimum – 0.2 %/min at 49 °C to a maximum – 1.7 %/min at 349 °C (16 minutes of testing). However, the decrease in mass observed at a given temperature is about 10 % (mass.) And is probably associated with the decomposition and evaporation of the organic part of the sample and moisture. With a further increase in temperature, the rate of decrease in mass decreases, and at 564 °C, it reaches zero. Thus, under high-temperature exposure, the studied material does not exhibit properties characteristic of flammable and combustible materials. The study results of the sample's behaviour confirm the non-combustible properties of the OGNEZA-M-KOR (NG) sealant declared by the manufacturer.

Analyzing the DTG curve of the fire seal OGNEZA-M-KOR (Figure 4), the following conclusions can be drawn. Starting from 15 minutes of thermal testing, a noticeable decrease in the mass of the test sample is observed, which reaches a maximum by 19.5 minutes at 341 °C (6.1 %/min). After reaching a temperature of 571 °C at the 29-th minute of research, the OGNEZA-M-KOR sample does not lose weight, demonstrating non-combustible properties.

The DTG curve of the fire-retardant material «OGNEBASALT» PMBOR (Figure 5) indicates an insignificant loss of sample weight over time – from 1.2 %/min at 337 °C to 3.5 %/ min at 388 °C. In the time interval 15.0-22.5 minutes, the rate of change in the mass of the sample is insignificant. After 27 minutes of exposure to temperature, the sample does not change up to 1000 °C. A sample of fire-retardant material «OGNEBASALT» PMBOR demonstrates non-combustible properties.

In fig. 6, the maximum rate of weight change – 11.9 %/min of the fire-retardant material OGNEZA-GT was observed at the 17-th minute of testing at a temperature of 367 °C. The duration of the process of changing the mass at a high rate is about 9 minutes and is probably related to the burnout of the organic part of the sample. A further increase in temperature is not associated with a noticeable decrease in the mass of the material.

The weight loss of test specimens of fire retardant materials upon heating (TG) is shown in Table 1.

Table 1. Characteristic of reducing the mass of samples of fire-retardant materials under thermal exposure

<table>
<tr><td></td><td colspan="10">Weight loss of samples,% (upon reaching temperature, °C)</td></tr>
<tr><td></td><td>10</td><td>20</td><td>30</td><td>40</td><td>50</td><td>60</td><td>70</td><td>80</td><td>90</td><td>100</td></tr>
<tr><td rowspan="2">OGNEZA -LTU</td><td>301</td><td>352</td><td>449</td><td>552</td><td>631</td><td>842</td><td>889</td><td>935</td><td>Not achieved</td><td></td></tr>
<tr><td colspan="10">The overall weight reduction of the sample is 82 %.</td></tr>
<tr><td rowspan="2">OGNEZA -M-KOR (NG)</td><td>461</td><td colspan="9">No change in mass occurs when heated to a temperature of 1000 °C</td></tr>
<tr><td colspan="10">The overall reduction in sample weight is 11 %.</td></tr>
<tr><td rowspan="2">OGNEZA -M-KOR</td><td>320</td><td>356</td><td>442</td><td colspan="7">Starting from a temperature of 555 °C, there is no change in mass.</td></tr>
<tr><td colspan="10">The overall reduction in sample weight is 37 %.</td></tr>
<tr><td rowspan="2">«OGNEB ASALT» PMBOR</td><td>395</td><td>481</td><td colspan="8">Starting from a temperature of 559 °C, no change in mass occurs.</td></tr>
<tr><td colspan="10">The overall reduction in sample weight is 21 %.</td></tr>
<tr><td rowspan="2">OGNEZA -GT</td><td>262</td><td>338</td><td>364</td><td>382</td><td>516</td><td>888</td><td colspan="4">No mass loss occurs.</td></tr>
<tr><td colspan="10">The overall reduction in sample weight is 64 %.</td></tr>
</table>

Based on the data in Table 1, it can be concluded that for the loss of, for example, 10 % of the mass, the test samples require different temperatures – from 262 °C to 461 °C. OGNEZA-M-KOR (NG) shows the highest resistance to temperature with a total weight loss of only 11 %. «OGNEBASALT» PMBOR, OGNEZA-M-KOR, demonstrates high resistance to temperature.

The least resistant to the temperature of OGNEZA-LTU, in which almost complete combustion of the sample occurred: the weight reduction was 82 %. The change in the rate of weight reduction is reflected in the DTG curve of the sample, which recorded a large number of peaks in the temperature range from 271 to 460 °C. The experiment has established the swelling of the OGNEZA-LTU sealing tape, which can cause weight loss. The sample exhibits fire-retardant properties, expanding many times when heated in volume and thereby preventing the propagation of combustion.

OGNEZA-GT sealant is a two-layer material consisting of dense and fibrous parts. After exposure to temperatures up to 1000 °C, the fibrous structure of the material is preserved, ensuring the integrity of 36 % of the sealant structure. The fire-retardant properties of this sample are probably associated not with the resistance to ignition but with the expansion of the non-combustible part in the volume, similar to the OGNEZA-LTU sealing tape.

4 Conclusions

1. The rates of weight loss of the investigated fire-retardant materials have been studied. A high rate of weight loss was established for OGNEZA-LTU samples (20.5 %/min at 335 °C) and OGNEZA-GT (11.9 %/ min at 369 °C). A low rate of weight loss was noted in the samples OGNEZA-M-KOR (NG) – 1.7 % /min at 349 °C, OGNEZA-M-KOR – 6.1 %/ min at 341 °C, «OGNEZALT» PMBOR – 3,5 %/min at 388 °C. Time intervals of weight loss have been determined: all tested samples have maximum weight loss in the range of 15-20 minutes.
2. The temperature ranges of the maximum rate of weight loss have been determined: 349 - 388 °C.
3. The best heat-resistant properties were shown by OGNEZA-M-KOR (NG), «OGNEBA-ZALT» PMBOR, OGNEZA-M-KOR.

The research results can be used to substantiate the choice of fire protection in buildings of various functional classes of fire hazard.

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